

Direct measurement and calculation of rubber bulk modulus by piston-cylinder method using conventional mechanical testing equipment

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ABSTRACT

This article describes a method to directly measure the bulk modulus of rubber using a piston-cylinder type apparatus. The function of the testing apparatus is to reliably transfer load to the water inside the vessel and allow for the accurate and continuous measurement of volumetric displacement as the contents are compressed. During the test, load is applied to a piston-cylinder containing the rubber test sample and water using a conventional mechanical testing load frame. Load and the travel of the piston are recorded by a load cell and extensometer, respectively. A brass or stainless steel plug of the same dimensions as the test sample is tested as a control sample to isolate the compliance of the system, including the compressibility of water and compliance of the o-rings which seal the piston-cylinder system. The results from this control test are used to decouple the compliance of the system from the sample test results. Load-linear displacement data are mathematically converted to pressure-volumetric displacement data, from which bulk modulus is calculated. The test apparatus and method are validated using the known bulk modulus of water. Finally, the bulk moduli for two similar rubbers are evaluated. The validity of the results and limitations of the device are discussed.

1. Introduction

Bulk modulus has been shown to be an important consideration in engineering, especially when the primary loading is compression [1]. However, existing mechanical properties databases for rubbers rarely report bulk modulus, despite its value to engineering design. This is because the bulk modulus of rubber (κ) has been very difficult to determine experimentally, owing to the difficulty of measuring volumetric deformation accurately under a hydrostatic stress. One definition of bulk modulus is $\kappa = \frac{\Delta P}{\Delta V/V_0}$, where ΔV is the change in volume due to a change in pressure ΔP and V_0 is the initial volume of the sample. For solids, typically the bulk modulus is calculated by Eq. (1) where E is the modulus of elasticity and ν is Poisson's ratio measured by uniaxial testing.

$$\kappa = \frac{E}{3(1 - 2\nu)} \quad (1)$$

However, for rubbers, κ is highly sensitive to ν in this relationship. The relationship, displayed in Fig. 1 shows $\frac{\kappa}{E}$ as it relates to ν over the range of 0.48 to 0.50, which is typical for rubbers. In this range, κ reaches a minimum of 8.33E for $\nu = 0.48$ and increases rapidly toward ∞ as ν approaches 0.50. Therefore an accurate evaluation of κ requires that ν be measured with extreme precision, not typically available with conventional mechanical testing equipment. It is worth

noting that the inverse relationship with regard to sensitivity is also true. Experimentally determined bulk modulus and Young's modulus with moderate precision will yield Poisson's ratio of high precision [2]. However, other researchers have argued that E and κ may arise from different inter-molecular mechanisms in rubbers [3] and therefore the validity of relating E , κ , and ν for rubbers by Eq. (1) should be examined. Additional difficulties in evaluating bulk modulus of rubbers arise from complex stress softening phenomena such as the Mullins effect [4]. In this regard, a method to determine bulk modulus by hydrostatic compression, such as used in this work, is ideal because it is not susceptible to appreciable stress softening [5].

To avoid the uncertainty of evaluating κ by applying Eq. (1) to experimentally determined E and ν , various attempts have been made to determine κ directly. Usually these measurements have been made under *quasi*-hydrostatic stress rather than true hydrostatic conditions due to the difficulty in accurately measuring volumetric deformations under true hydrostatic stress conditions [2,6,7].

In this article, a method is described to measure bulk modulus under true hydrostatic conditions (neglecting the very small head pressure gradient due to gravity) using conventional mechanical testing equipment and a simple piston-cylinder type apparatus. The results give repeatable figures with reasonable precision, which was validated using

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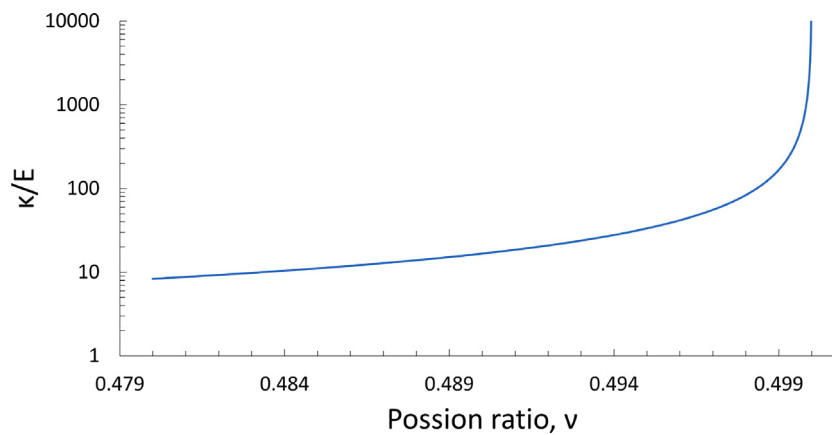


Fig. 1. $\frac{K}{E}$ as a function of Poisson's ratio, ν , in Eq. (1) over the typical range for rubbers.

the well-known compressibility of water as a standard. The apparatus and method developed herein is broadly applicable to any mechanical testing facility using conventional testing load frames and extensometry equipment. In other works, there have been discussions on the importance of being precise in terminology when reporting values for bulk modulus. For instance, a bulk modulus can either be secant or tangent and either isothermal or isentropic. Each combination of these variables give the four basic types of bulk modulus: isothermal secant, isothermal tangent, isentropic secant, and isentropic tangent [8]. The results reported in this paper are tangent approximations and are in between isothermal and isentropic.

2. Design and operation

2.1. Mechanical design

The configuration of the physical apparatus is shown in Fig. 2, which consists of the (1) Piston, (2) Vessel, and (3) Plug. Prior to operating the apparatus, the (3) plug is inserted into the (2) vessel; the (3 A) plug o-ring will seal the water in at the bottom of the vessel. Water is poured into (2C) the center bore of the vessel and sample inserted such that the sample and (2B) purge hole are fully submerged. The water and sample space is de-aerated under vacuum to remove any air bubbles trapped at the surfaces of the bore and sample. The piston is inserted into the vessel and pressed until the (1C) o-ring begins to seal against the face of the water at the purge hole. Any air trapped below the o-ring is forced out of the sample space through the purge hole before the o-ring seals. At this point the apparatus is prepared for the test. The (1B) secondary o-ring on the piston keeps the piston concentric with the center bore of the vessel during the test. The diameters at the (1 A) top of the piston and the (2 A) collar of the vessel are equal by design to facilitate the continuous displacement measurement between these two features by non-contact extensometry.

Non-contact extensometry requires two reference measurement points, one on the piston top and one on the vessel collar, to be at the same distance from the extensometer for accurate measurement. Alternative methods of measuring displacement such as a physical extensometer may be applied for the same result by fixing the extensometer arms the same points on the apparatus. The vessel and plug were constructed from 316/316L stainless steel. The piston was constructed from Hastelloy X to prevent galling across the small clearance between the piston and the vessel during testing. The O-rings used were Buna-N (NBR) material dash size 312 and of hardness 90 A. The vessel was qualified for service up to 36.5 KSI (251.7 MPa) using ASME Boiler Code Division VIII, Section 8, page 69 [9]. Detailed schematics of each component and calculation of pressure rating are published in Appendix A and Appendix B, respectively.

2.2. Testing procedure

During a test, force was applied to the top of the (1) piston to pressurize the water-filled void, applying a hydrostatic stress state to the sample. The force was applied via a mechanical testing load frame (MTS Systems, Eden Prairie, MN), which was operated in position control at 0.15 mm/min. Applied force was measured by a 10,000 lbf (45 kN) load cell. The displacement of the piston is measured via non-contact extensometry (Epsilon ONE, Epsilon Technology Corp, Jackson, WY) by relating the positions of the (1 A) top of the (1) piston and the (2 A) collar of the (2) vessel. Data was recorded at 10 Hz. The orientation of the components during a test are shown in Fig. 3. A photograph of a test in progress is shown in Fig. 4.

3. Data analysis

Calibration was accomplished with either a stainless steel or brass specimen which is considered to be incompressible relative to water and rubber; the bulk moduli of steel and brass are over both 100 GPa, which is two orders of magnitude greater than that of water and the rubber specimens examined. The data from the calibration test plotted as load vs. displacement has a slope which can be used to derive the compliance of the system due to the compressibility of water and deformation of o-rings, piston, and vessel. An example of this calibration test data is shown in Fig. 5 along with raw data collected from a rubber test specimen.

Under the force of the test frame all the components of the piston-cylinder apparatus system interact with each other in series such that each of the components experience the same pressure. Under this pressure, each component compresses by a volume proportional to pressure applied, the volume of each component, and the compressibility of each component. The total compression of the system is equivalent to the sum of the compressions of each component. This is very similar to the way the displacements of springs in series are additive and independent of each other when a force is applied. Therefore, the same mathematical concept which is used to assess the spring constant of a system of springs in series can be used to assess the relationship between compressible components under pressure and interacting in series. Consequently, Eq. (2) shows how this concept can be used to isolate the displacement attributable to the compression of the specimen from that of the rest of the system. In the piston cylinder apparatus system, the most impactful of the components which make up the compressible volume of the system are assumed to be the sample, the water surrounding the sample, and the o-rings which seal the system.

To remove the compliance of the system from the test data, the difference between the reciprocal slopes of the control and test runs was found, then inverted to give the slope of the compliance-corrected

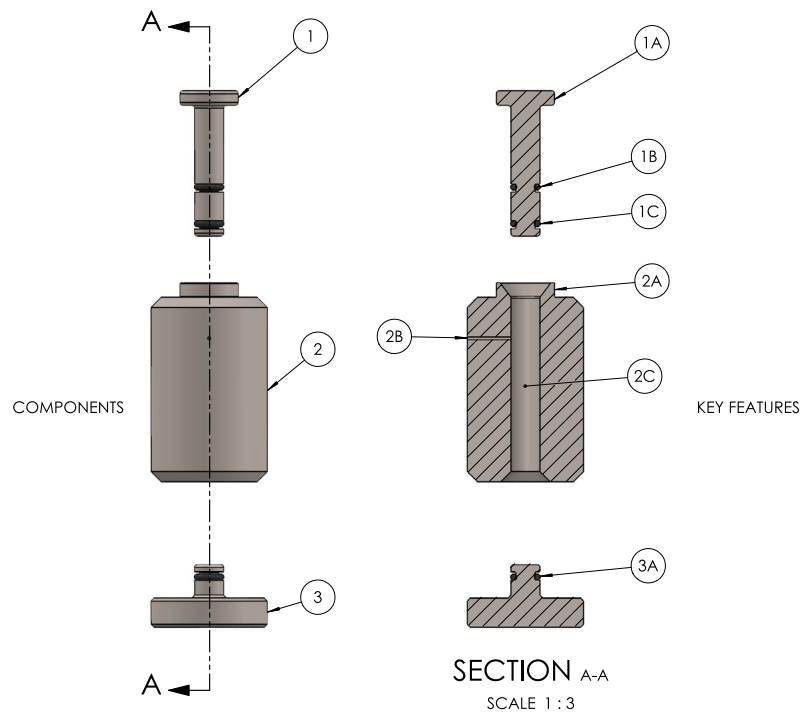


Fig. 2. Testing apparatus components and key features. Components include: (1) piston, (2) vessel, and (3) plug. Key features include (1 A) top of the piston, (1B) secondary piston o-ring, (1C) sealing piston o-ring, (2 A) vessel collar, (2B) purge hole, (2C) center bore of the vessel, (3 A) sealing plug o-ring.

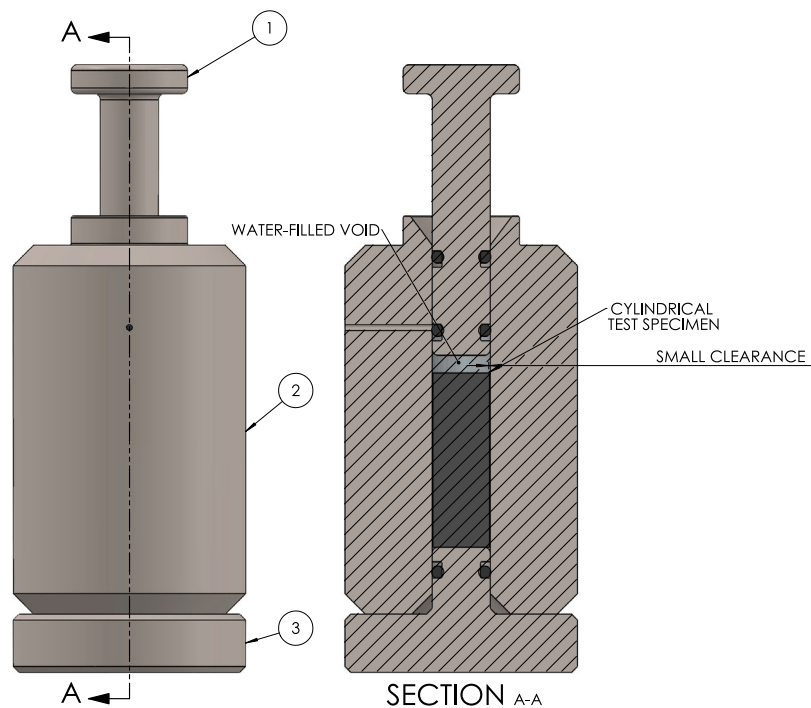


Fig. 3. Testing apparatus configured at the beginning of a test.

test in units of $\frac{Force}{Length}$. The slopes are obtained by linear fit only in the linear portion of the force–displacement curve, such as that shown in Fig. 5, using identical force bounds for the control and sample data (rather than displacement bounds). No displacement offset to the raw data is required. Secant slope or linear slope fit over an approximately linear range can be used if the data does not have significant noise. However, linear fit is recommended to reduce error due to noise. The mathematical process is shown in Eq. (2) where $(\frac{dx}{dF})_{system+sample}$ is the

reciprocal slope of the raw data from the test run, $(\frac{dx}{dF})_{system}$ is the reciprocal slope of the raw data from the control run and $(\frac{dF}{dx})_{sample}$ is the compliance-corrected slope of the test data. To determine the slope at a pressure a linear fit was applied to the load and displacement data in a 1000 PSI (6895 KPa) window around the pressure in question. For example, to get a slope for 2000 PSI (13790 KPa) a linear fit was applied to the load vs displacement data from the load value corresponding to 1500 PSI (10342 KPa) to the load value corresponding



Fig. 4. Photo taken during testing. Load is being applied by a hydraulic load frame. Green lighting is from the non-contact extensometer at the right. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

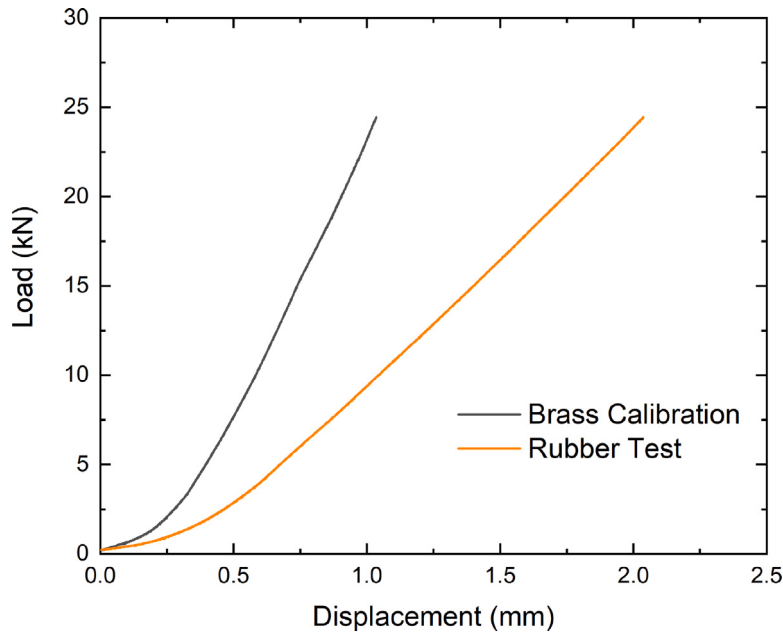


Fig. 5. Raw data from a calibration test using a brass specimen and a test of a rubber specimen.

to 2500 PSI (17237 KPa).

$$\frac{1}{\left(\frac{dx}{dF}\right)_{\text{system+sample}} - \left(\frac{dx}{dF}\right)_{\text{system}}} = \left(\frac{dF}{dx}\right)_{\text{sample}} \quad (2)$$

In this apparatus, ΔV is measured by the linear displacement dx of the piston head with consideration of the cross-section of the sample space, which is the vessel's bore, A_{vessel} , such that $\Delta V = (x - x_0)A_{\text{vessel}}$. Thus, the bulk modulus of the sample can be calculated by Eq. (3), where V_i is the instantaneous volume of the sample at a particular pressure during the test. V_i was found for each sample by taking the difference in piston displacements between the control run (brass or stainless steel sample) and the test run at a particular pressure and relating that displacement to a volumetric change in the sample. The mathematical equation for this is shown in Eq. (4), where $x_{i,\text{test}}$ and $x_{i,\text{control}}$ are the linear displacements of the piston relative to the vessel during the test run and control run, respectively, at a particular pressure.

$$\kappa = \frac{\Delta P}{\Delta V/V_0} = \left(\frac{dF}{dx}\right)_{\text{sample}} \times \frac{V_i}{(A_{\text{vessel}})^2} \quad (3)$$

$$V_i = V_0 - (x_{i,\text{test}} - x_{i,\text{control}})A_{\text{vessel}} \quad (4)$$

4. Results and discussion

4.1. Validation using water

Due to the difficulty in accurately testing the bulk modulus of polymers and the lack a polymeric standard with known bulk modulus, water was used as a validating test specimen since it has a widely accepted compressibility. This was accomplished by running a compliance test with a brass test specimen, then testing the vessel filled entirely with water (*i.e.*, no test specimen.) Then the analysis described in Section 3 was applied to obtain the measured bulk modulus of water. Fig. 6 compares the results obtained using this apparatus to those available in literature, which were measured using a sound velocity technique [10]. The measured bulk modulus of water is in fair agreement with the reference sound velocity measurements when measured around 15

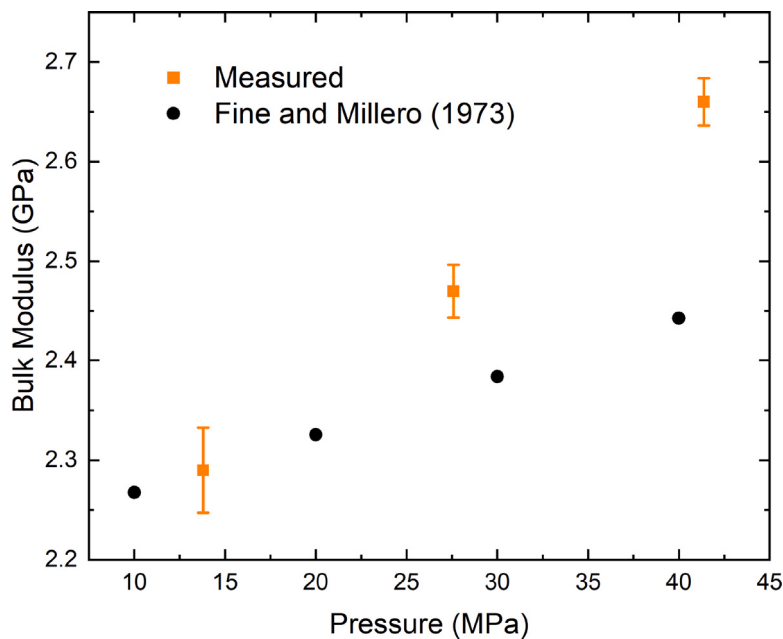


Fig. 6. Bulk modulus of water measured using the apparatus compared to reference data at room temperature [10].

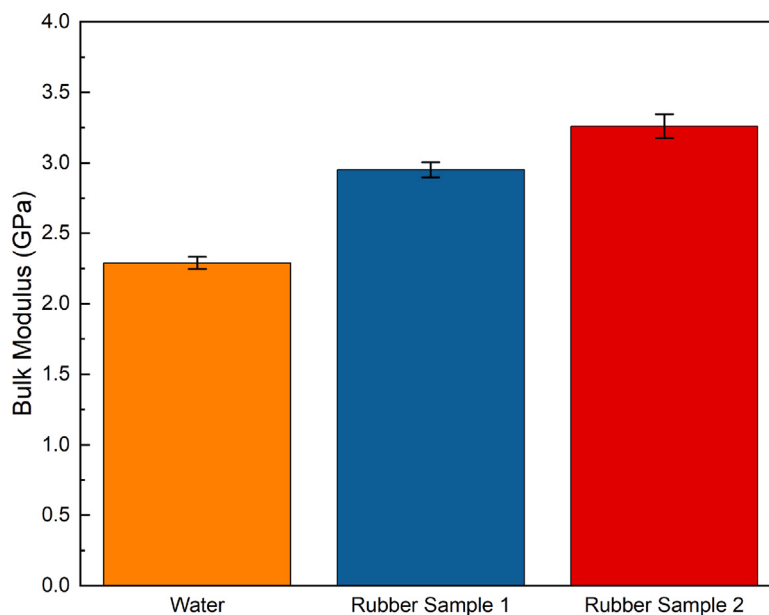


Fig. 7. Bulk modulus evaluated at 2000 PSI (13.79 MPa) for water and two rubbers with different durometer hardness. Values for each rubber represent the average of four samples; error bars represent one standard deviation.

MPa. However, the discrepancy between the data sets increases with pressure such that at about 40 MPa, the measurements differ by 10%.

Due to the trend observed in Fig. 6, the apparatus discussed in this paper may be most accurate at lower pressures, such as those below 15 MPa. At higher pressures, the measured bulk modulus is elevated when compared to sound velocity measurements [10]. It is believed that this deviation is due to a few different types of phenomena. One such phenomenon is that the pressurization of liquid water adiabatically and isentropically causes a rise in temperature [11] that causes a consequent increase in bulk modulus, as is demonstrated in Ref. [10]. Additionally, the energy required to overcome friction (and turbulence in the water during pressurization) is not isentropic and increases the internal energy of the water, further elevating temperature [11]. Separately, the frictional force associated with the movement of the piston

due to the water-tight seal between the piston and the vessel provided by the sealing o-ring may contribute to the observed deviation along with deformation of the o-rings. As pressure increases in the vessel, the sealing o-ring will further extrude into the clearance gap between the piston and the inner wall of the vessel, presumably increasing the piston's frictional resistance to movement relative to the vessel. It is believed that this frictional resistance is negligible at low pressures such as 15 MPa but may become significant at higher pressure. The data reported in the referenced study [10] is not susceptible to the same sources of error because that study uses a completely different test method, namely through sound speed measurements. It is worth noting at this point that sound velocity methods for measuring the bulk modulus of water used as a standard for comparison do not appear in literature to have been successfully applied to rubbers.

Other means to improve the data given through the piston-cylinder method may include decreasing the speed of load application and decreasing the diameter of the vessel's bore. Initial testing was done at 0.3 mm/min load frame speed but produced aberrant non-monotonic results which deviated from the expected nearly linear plots of force with respect to displacement. However, slowing the rate to 0.15 mm/min produced a relatively linear plot. Alternatively, a pressure transducer could be installed so the pressure could be reported directly and thereby subvert the errors associated with force measurements on the piston. Also, by decreasing the diameter of the vessel's bore and maintaining the initial volume of the sample, an increase in the stroke distance during a test would be achieved. This would yield a higher resolution in ΔV from Eq. (3), which may improve precision in κ .

4.2. Rubber test specimens

Rubber specimens were cut from industrial-grade rubbers commonly used for primary suspension pads in railcar suspension systems and tested to evaluate the applicability of the apparatus to commercial rubber. The results found for bulk modulus at 2000 PSI (13,790 kPa) are shown in Fig. 7 along with the results for the water found in validation tests. Two similar rubbers were tested: Rubber Sample 1 had a nominal Shore D durometer hardness of 63 and Rubber Sample 2 had a nominal Shore D durometer hardness of 55. Durometer is a surface measurement and therefore may not be a representative bulk property. The bulk modulus of these rubbers at 2000 PSI (13,790 kPa) were determined to be 2.95 ± 0.05 GPa and 3.26 ± 0.08 GPa, respectively. These results are reasonable when compared to the published bulk modulus of similar rubbers found through other means [12].

5. Conclusions

A bulk modulus mechanical property testing apparatus and calculation was developed in this work. Water was used as a validating test specimen because of its well-established values for compressibility, which is the inverse of bulk modulus. The results of validation

testing closely matched the published bulk modulus for water at room temperature and 2000 PSI (13.79 MPa). Divergence from previous and corroborated values was observed at pressures of 4000 and 6000 PSI, likely due to excessive friction and o-ring deformation, and perhaps thermodynamic phenomena. Tests of industrial-grade rubbers with different durometer hardness yielded reasonable values for bulk modulus as compared to similar rubbers tested through other means. This apparatus provides a readily available means to directly evaluate bulk modulus of rubber and other polymers using conventional mechanical testing equipment.

Declaration of competing interest

The authors declare that they have no competing financial or personal relationships that may be perceived as influencing the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgments

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Appendix A. Apparatus dimensions

A.1. Vessel dimensions

See Fig. A.8

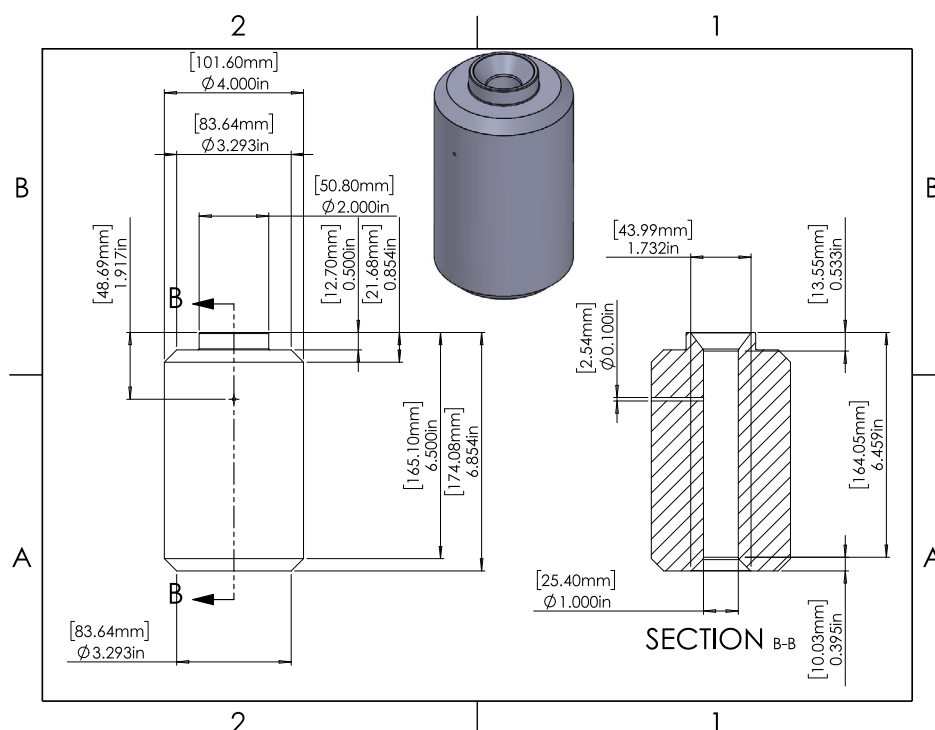


Fig. A.8. Schematics of vessel design.

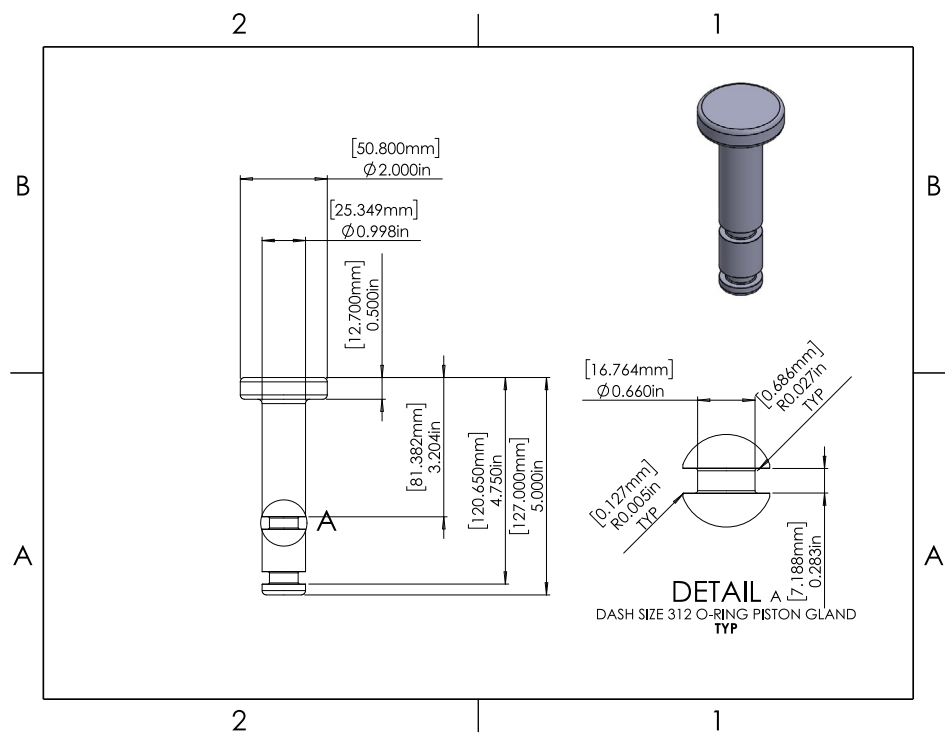


Fig. A.9. Schematics of piston design.

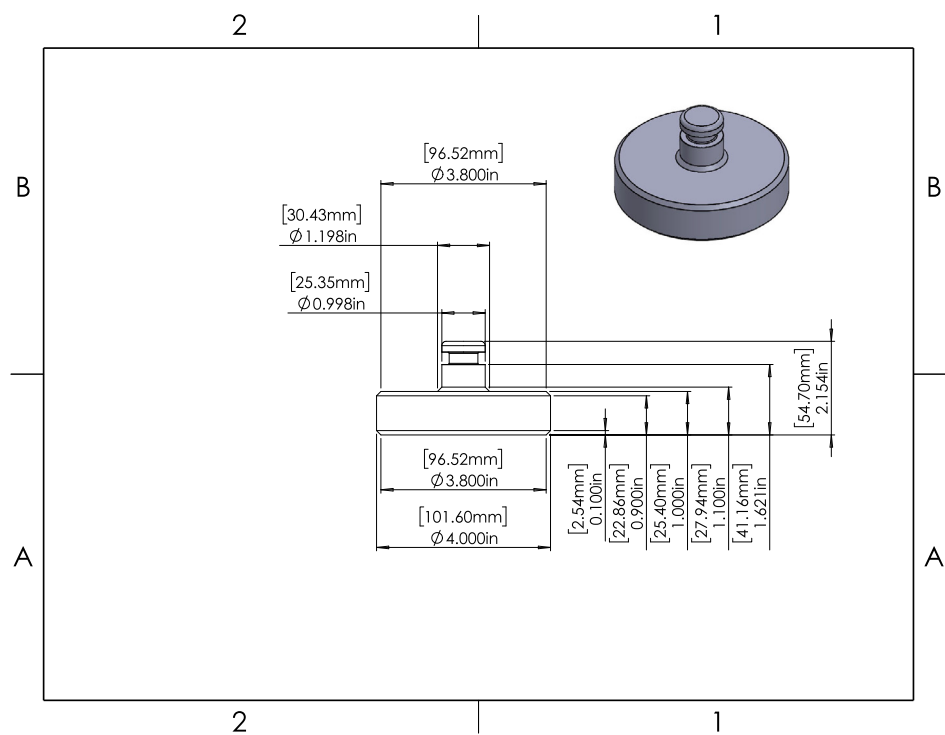


Fig. A.10. Schematics of vessel plug. For details on o-ring gland, see [Appendix A.2](#).

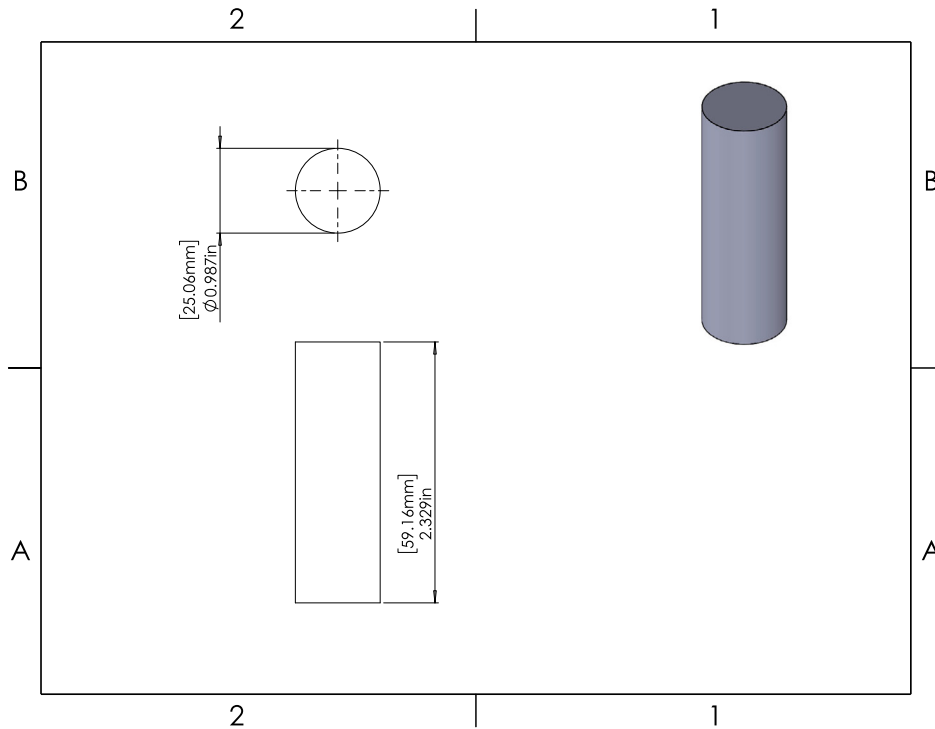


Fig. A.11. Approximate sample dimensions. The sample and the dummy plug should have as identical dimensions as are reasonably possible.

A.2. Piston dimensions

See Fig. A.9

A.3. Plug dimensions

See Fig. A.10

A.4. Sample dimensions

See Fig. A.11

Appendix B. Supplemental calculations

B.1. Vessel pressure rating qualification using the ASME boiler code, section 8, division 3, page 69

Material Properties:

$$S_y = 30 \text{ KSI (207 MPa)}$$

$$S_u = 75 \text{ KSI (517 MPa)}$$

K_{ut} = factor of upper limit for hydrostatic test pressure

$$= 0.95 \text{ for } S_y/S_u \leq 0.7$$

$$\rightarrow K_{ut} = 0.95$$

Geometric Properties:

$$D_O = 4 \text{ in (101.6 mm)}$$

$$D_I = 1 \text{ in (25.4 mm)}$$

$$Y = D_O/D_I$$

$$\rightarrow Y = 4$$

KD-221.2 For Open-End Cylindrical shell for $Y > 2.85$ and Closed-End Cylindrical Shell for All Y Values:

$$P_D = \min([0.924 K_{ut}(S_y) \ln(Y)],$$

$$[\frac{1}{3}(S_y + S_u) \ln(Y)])$$

$$\rightarrow P_D = \min(36.5 KSI, 48.5 KSI) = \min(251.7 \text{ MPa}, 334.4 \text{ MPa})$$

$$\rightarrow P_D = 36.5 \text{ KSI (251.7 MPa)}$$

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